

Creep-Rupture Behavior of a Developmental Cast-Iron-Base Alloy for Use Up to 800 °C

Robert H. Titran and Coulson M. Scheuermann
National Aeronautics and Space Administration
Lewis Research Center

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National Aeronautics and Space Administration
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SUMMARY

As part of the DOE/NASA Stirling Engine Systems Project, a promising iron-base cast alloy is being developed under contract DEN 3-282 with the United Technologies Research Center (UTRC), designated NASAUT 4G-A1. Its nominal composition, in percent by weight, is Fe-15Mn-15Cr-2Mo-1.5C-1Nb-1Si.

This report presents the results of a study of this alloy, 4G-A1, performed at the NASA Lewis Research Center to determine its creep-rupture properties. The alloy was studied in the directionally solidified (DS) form with a 650 °C/100 hr anneal recommended by UTRC to optimize properties and in the investment-cast (IC) form with either a 760 °C/20 hr anneal recommended by UTRC to optimize properties, or a solution anneal of 790 °C/20 hr followed by a simulated brazing cycle of 1065 °C/15 min + a heat treatment of 760 °C/16 hr + 650 °C/16 hr.

Alloy 4G-A1 exhibited typical 3-stage creep response under all conditions tested. The most creep resistant condition was the DS material. This condition compares very favorably to the prototype (HS-31) and prime candidate (XF-818) alloys for the automotive Stirling engine cylinder/regenerator housing.

INTRODUCTION

An advanced materials technology program at NASA Lewis Research Center has been supporting the DOE/NASA Stirling Engine Systems Project. This program is directed towards meeting the high temperature components requirements of Stirling engines. One of the most critical components is the heater head, consisting of cylinders, regenerators and tubing. The major high temperature materials requirements have been described in assessments of Stirling engine materials technology (refs. 1 and 2).

ENGINE REQUIREMENT AND ALLOY SELECTION

The MOD 1A Stirling automotive engine design criteria is based upon 3500 hr of engine operation under a combined 55 percent urban/45 percent highway driving cycle. The heater head tubes will experience a mean temperature of 820 °C and the cylinder-regenerator housings a maximum temperature of 775 °C. The hydrogen working fluid will have an equivalent average pressure of 7.2 MPa. The design criteria include a safety factor of 1.5; thus the design stress needed for a target rupture life of 3500 hr would be about 28 MPa for heater head tubes and about 119 MPa for cylinder and regenerator housings.

(ref. 14). It has been found in recent engine operation that no cylinder or regenerator housing failures occurred by creep-rupture, but failure did occur by fatigue. Therefore, it is believed that this creep-rupture requirement is very conservative.

A promising alloy resulting from this program is being developed under contract DEN 3-282 with the United Technologies Research Center. It has been designated NASAUT 4G-A1. The nominal composition of this alloy (in weight percent) is Fe-15Mn-15Cr-2Mo-1.5C-1Nb-1Si. The alloy is austenitic and is strengthened by solid solution strengtheners and carbide phases (ref. 3).

For an automotive application with a conservative volume at 400 000 engines per year, estimates show that current cobalt production could not meet the demand of this potential single use of cobalt. Since it contains no cobalt or nickel, its cost is reduced considerably and this new nickel and cobalt-free alloy is more attractive than the cobalt alloy, X-40 (HS-31) used in initial prototype engines.

A study was made of the creep-rupture properties of this alloys at NASA Lewis by the authors. This report describes the results of this study.

MATERIAL

The alloy, NASAUT 4G-A1, was determined to possess the best combination of properties of the experimental alloys investigated by UTRC in this program. To a limited extent, its mechanical properties were evaluated at UTRC in both the conventional investment cast (IC) and directionally solidified (DS) configurations. The alloy compared favorably with those being used for the engine development program (alloys X-40 (HS-31) and XF-818) (refs. 3 to 9).

Tensile bars of both IC- and DS-cast alloy were obtained from UTRC of the same stock as used in their testing program. Specimens tested in this study were given pre-test heat treatments as shown in table 1. Baseline DS specimens were given an anneal of 650 °C/100 hr in air and air cooled while the IC specimens were annealed 760 °C/20 hr in vacuum. This was believed by UTRC to yield optimum properties for the alloys for each respective casting method. A later heat treatment, used on the investment-cast specimens, was devised due to the need to braze heater tubes to the heater head casting for engine assembly. The heat treatment consisted of a solution anneal at 790 °C/20 hr followed by a simulated braze cycle of 1065 °C/15 min + 760 °C/16 hr + 650 °C./16 hr. The final two temperature-time combinations are required for the tube alloy, CG-27, which is a precipitate-strengthened alloy. Figure 1 shows the microstructures of the alloy following each of these heat treatments.

EXPERIMENTAL PROCEDURE

Compositions of the material investigated in this study are listed in table II. The DS material was supplied in the form of 1.3 cm diameter by 26 cm long bars. Creep-rupture test specimens were machined with the longitudinal test direction parallel to the solidification direction. The solidification direction was reported by UTRC to be nominally <100>. The IC material was

supplied in the form of investment cast threaded test specimens as shown in figure 2.

Tensile creep-rupture tests were conducted in air in accordance to ASTM E139. Constant load creep-rupture tests were conducted in the 760 to 900 °C region in conventional beam-loaded machines. Strain measurements during creep were determined from the movement of an extensometer attached to the reduced section of the specimens and converted to an electrical signal by means of a linear variable differential transformer. Test temperatures were measured by Pt-Pt13Rh thermocouples attached to the specimen reduced gage section.

RESULTS AND DISCUSSION

Typical creep curves for the 4G-Al alloy in the DS and IC conditions and having their optimum heat treatments are shown in figure 3. The 4G-Al material is noted to exhibit classical creep behavior, i.e., three stage creep. Figure 3 shows the smooth, almost parabolic primary creep stage, followed by the secondary steady-state stage which determines the minimum creep rate and into the third stage leading to rupture.

Base-line creep rupture data at 760 to 900 °C from tests of the optimum heat treated alloys results from similar tests on simulated braze cycled materials are summarized in tables 3 to 5. The effects of stress on the minimum creep rate and on the rupture life of the three NASAUT 4G-Al material conditions at the creep-rupture test temperatures are given in figures 4 and 5.

Temperature-compensated analysis of the minimum creep rate (ϵ_m , sec⁻¹) and the time to rupture (t_R , hr) as a function of stress were performed using a form of the Orr-Sherby-Dorn (ref. 10) power law relationship. Using multiple linear regression analysis, activation energies for creep were determined for each condition based on minimum creep rates as well as rupture lives. These activation energy values are listed in table 6 along with stress exponents and constants for the following relationships:

$$\ln \dot{\epsilon}_m = \ln k_1 + n_1 \ln \sigma + \frac{Q_1}{RT} \quad (1)$$

$$\ln t_R = \ln k_2 + n_2 \ln \sigma + \frac{Q_2}{RT} \quad (2)$$

where ϵ_m is the minimum creep rate (sec⁻¹), t_R is rupture life (hr), k_1 and k_2 are constants, n_1 and n_2 are stress-term exponents, σ is stress (MPa), R is gas constant (8.314 J/K-mol), T is absolute temperature (K), and Q_1 and Q_2 are apparent activation energies (KJ/mol) (refs. 11 to 13). Equations (1) and (2) along with the values in table 6 are useful in predicting rupture lives and minimum creep rates within the present test conditions, but the reliability of extrapolations beyond these conditions is not recommended.

It is noted in figure 4 that all conditions tested are plotted with a single slope for ϵ_m versus stress suggesting that in the temperature and stress region studied only one creep mechanism was operative. The minimum creep rate (ϵ_m) equation for each condition is given in table VI by the coefficients (Q_1 , n_1 , and $\ln K_1$) and is valid for the stress and temperature range

studied. From figure 4 it is also noted that the most creep resistant condition is the directionally solidified condition followed by the investment cast materials. For example, at 775 °C and a 10^{-8} creep rate the stress levels range from about 200 MPa, to 165 MPa, to 145 MPa for the DS, and for the investment cast annealed and braze cycled materials, respectively.

In figure 5 the same singular slope for each test condition is noted for creep-rupture life (t_r) versus stress. The creep-rupture time (t_r) equation for each condition is given in table VI by the coefficients (Q_2 , n_2 , and $\ln K_2$) and is valid for the stress and temperature range studied. From figure 5 it is noted that the directionally solidified condition has the greater stress-rupture strength. For example the 1000-hr rupture life at 775 °C for the DS material occurs at about 200 MPa while for the investment cast material it is about 150 MPa and for the braze cycled material is 130 Mpa.

Elongation and reduction in area measurements following rupture generally indicate increased ductility with increased stress at constant temperature, over the range of loads applied in this study and agree with a previous creep-rupture study (ref. 14) of iron-base superalloys. Tables III and IV show that the percent reduction in area (RA) and percent elongation (EL) of the NASAUT 4G-Al material is reduced when the casting method is changed from DS to investment. The DS alloys generally show a RA of about 60 percent and a EL of about 17 percent whereas the investment cast condition only shows about a 6 percent RA and an EL of about 5 percent. The high temperature braze cycle heat treatment tends to restore a small percentage of the lost ductility of the investment cast material.

Fracture of NASAUT 4G-Al generally is of a ductile nature, although the carbide phases tend to crack and form the initiation sites for the ductile failure of the austenitic matrix. Visual inspection of the fractured specimens showed a roughly cup-cone type of failure. Metallography (fig. 6) also confirmed a ductile behavior of the matrix phase, giving an over all ductile fracture behavior, as indicated by the respectable ductility values of tables III to V. On-going studies will determine the effect of a brazing cycle on rupture ductility of DS material, longitudinal and transverse to the solidification direction.

In figure 7 the temperature dependence is shown for the extrapolated 3500 hr rupture strength of the NASAUT 4G-Al alloy, the prime candidate alloy XF-818, (ref. 14) and the prototype alloy HS-31 (ref. 14). The NASAUT 4G-Al alloy in the DS and in the investment cast condition both exceed the MOD 1A Stirling engine design criteria in the non-braze-cycled condition. The DS material is 35 percent stronger than the equiaxed investment cast material, 165 MPa versus 125 MPa at 775 °C. Subjecting the investment cast material to the simulated braze cycle resulted in about 12 percent loss in rupture strength. If we assume that the braze cycle heat treatment will have a similar effect on the DS material with a parallel reduction in rupture strength, the NASAUT 4G-Al in the DS condition is considered a highly acceptable alloy for Stirling engine applications.

CONCLUDING REMARKS

This study has shown that the casting techniques and high temperature brazing operations affect the creep-rupture properties of the NASAUT 4G-A1 alloy. The loss in strength due to investment casting plus additional braze-cycle heater tube heat treatment has been shown to be prohibitive in terms of potential engine application. If necessary, either engine design or modifications in alloy composition as well as heat treatment to improve or optimize creep-rupture strength could compensate for this strength loss. Fatigue, corrosion, and oxidation resistance are currently being investigated for the 4G-A1 alloy to further characterize the alloy for automotive Stirling engine applications.

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TABIF 1. - CASTING TECHNIQUES AND HEAT TREATMENT OF NASAUT 4G-A1
PRIOR TO CREEP/RUPTURE TESTING

Casting technique	Heat treatment
Directional solidified (DS)	650 °C/100 hr in air air cooled
Investment cast (A)	760 °C/20 hr in 10 ⁻² Pa vacuum - furnace cooled
Investment cast (A + BC)	790 °C/20 hr in 10 ⁻² Pa vacuum - furnace cooled 1065 °C/15 min vacuum furnace cooled 760 °C/16 hr vacuum furnace cooled 650 °C/16 hr vacuum - furnace cooled

TABLE II. - CHEMICAL ANALYSES OF NASAUT 4G-A1

	Element, wt %						
	Fe	Cr	Mn	Mo	Nb	Si	C
DS	66.7	14.3	11.7	1.9	0.9	1.0	1.5
Investment cast	63.7	14.4	14.1	2.0	.9	1.0	1.6

TABLE III. - CREEP RUPTURE DATA FOR DIRECTIONALLY SOLIDIFIED
NASAUT 4G-A1 ANNEALED AT 650 °C FOR 100 hr IN AIR AND
AIR COOLED PRIOR TO TESTING

Test temperature, °C	Test stress, MPa	Rupture time, hr	Minimum creep rate, sec ⁻¹	Time to 1 percent strain, hr	Percent RA	Percent EL
760	240	281.6	2.24x10 ⁻⁸	3.28	60	26
760	225	946.5	4.75x10 ⁻⁹	8.43	49	14
760	210	1264.4	4.78x10 ⁻⁹	16.45	59	16
760	180	^a 4520.4	6.10x10 ⁻¹⁰	^b 30.15	58	15
815	180	199.4	2.30x10 ⁻⁸	3.77	65	18
815	150	946.6	3.19x10 ⁻⁹	^b 191.08	66	21
815	140	^a 1645.3	1.77x10 ⁻⁹	94.53	67	19
815	125	^a 3273.2	9.62x10 ⁻¹⁰	691.93	55	14
900	103	170.5	1/70x10 ⁻⁸	81.05	70	15

^aPower interruption during test.

^bTreated as missing value in regression.

TABLE IV. - CREEP RUPTURE DATA FOR NASAUT 4G-A1 ALLOY
PRODUCED BY INVESTMENT CASTING. ANNEALED 760 °C for
20 hr AT 10^{-3} Pa; FURNACE COOLED PRIOR TO TESTING

Test temperature, °C	Test stress, MPa	Rupture time, hr	Minimum creep rate, sec^{-1}	Time to 1 percent strain, hr	Percent RA	Percent EL
725	220	676.0	1.28×10^{-8}	34.60	9	7
725	205	868.4	7.00×10^{-9}	60.44	9	7
725	190	1159.4	5.82×10^{-9}	200.08	5	4
775	180	213.3	3.16×10^{-8}	5.94	6	5
775	165	429.6	8.19×10^{-9}	58.94	3	2
775	150	1209.7	3.20×10^{-9}	536.72	13	6
775	140	1262.8	2.60×10^{-9}	581.39	3	2
825	130	274.2	-----	115.59	4	4
825	110	1039.1	-----	337.45	5	6
825	100	^a 844.1	-----	334.29	--	--

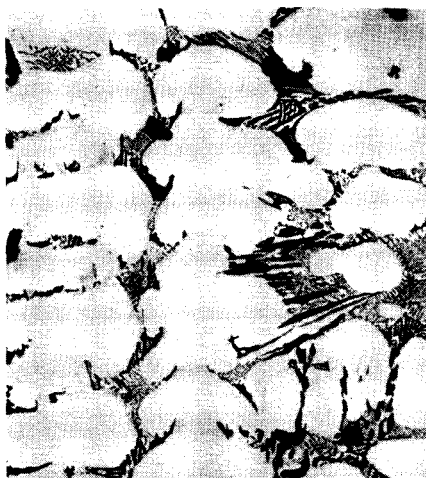
^aPower interruption during test.

TABLE V. - CREEP RUPTURE DATA FOR NASAUT 4G-A1 ALLOY PRODUCED BY
INVESTMENT CASTING. MATERIAL ANNEALED 790 °C for 20 hr AT 10^{-2} Pa
VACUUM THEN SIMULATED BRAZE CYCLE ANNEALED: 1065 °C - 15 min
PLUS HEATER TUBE SIMULATED HEAT TREATMENT OF 760 °C - 16 hr,
650 °C - 16 hr PRIOR TESTING FURNACE COOLED PRIOR TO TESTING

Test temperature, °C	Test stress, MPa	Rupture time, hr	Minimum creep rate, sec^{-1}	Time to 1 percent strain, hr	Percent RA	Percent EL
725	250	41.8	5.00×10^{-7}	1.44	38	21
↓	220	100.4	1.80×10^{-7}	7.63	29	17
	210	157.8	1.01×10^{-7}	7.86	14	12
	200	220.6	6.24×10^{-8}	13.42	15	9
↓	180	608.2	1.77×10^{-8}	153.94	11	8
775	180	52.6	2.36×10^{-7}	0.24	26	21
↓	160	304.6	3.93×10^{-8}	23.17	41	23
	150	752.1	1.10×10^{-8}	85.00	15	9
	150	361.6	2.55×10^{-8}	105.14	4	4
↓	140	556.3	9.22×10^{-9}	90.70	6	5
	136	1965.0	2.59×10^{-9}	663.10	20	8
	130	1140.6	2.95×10^{-9}	381.6	4	3
825	120	175.9	2.00×10^{-8}	45.18	3	4
↓	110	415.9	7.37×10^{-9}	178.01	3	4
	105	489.8	6.63×10^{-9}	224.22	2	2
	100	1145.1	-----	-----	2	5
↓	100	1810.8	1.88×10^{-9}	1127.13	--	10

TABLE VI. - MULTIPLE LINEAR REGRESSION ANALYSIS OF CREEP AND RUPTURE LIFE OF NASAUT 4G-A1

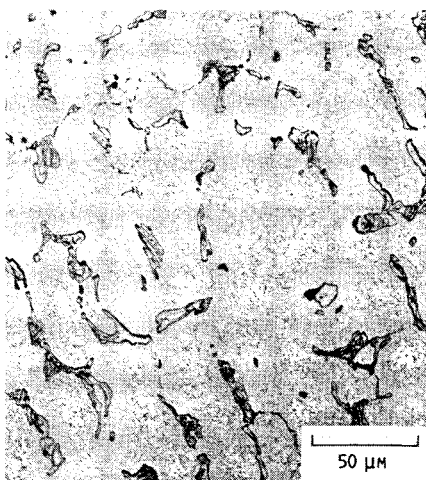
Material condition	Minimum creep rate data					Rupture life data				
	Number data points	Correlation coefficient, R^2	Q_1 , KJ/mol	n_1	$\ln K_1$	Number data points	Correlation coefficient, R^2	Q_2 , KJ/mol	n_2	$\ln K_2$
(DS) Directional solidified (ann:650 °C - 100 hr air)	9	0.94	-622	10.17	-1.41	9	0.97	567	-8.44	-13.74
(A) Investment cast (ann:760 °C - 20 hr vacuum	8	0.90	-377	8.98	-21.05	8	0.92	381	-6.79	-2.97
(A + BC) Investment cast (ann:790 °C - 15 min + 1065 °C - 15 min 760 °C - 16 hr 650 °C - 16 hr)	14	0.98	-450	11.73	-24.63	14	0.94	414	-8.65	1.44



DIRECTIONALLY SOLIDIFIED



CAST AND ANNEALED



CAST AND BRAZE CYCLED

FIGURE 1. - MICROSTRUCTURE OF HEAT TREATED NASAUT 4G-A1.

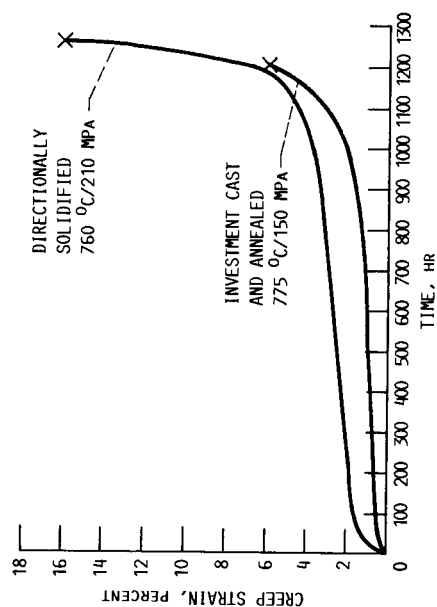


FIGURE 2. - CREEP RUPTURE CAST TEST SPECIMEN DESIGN. DIMENSIONS ARE IN MM.

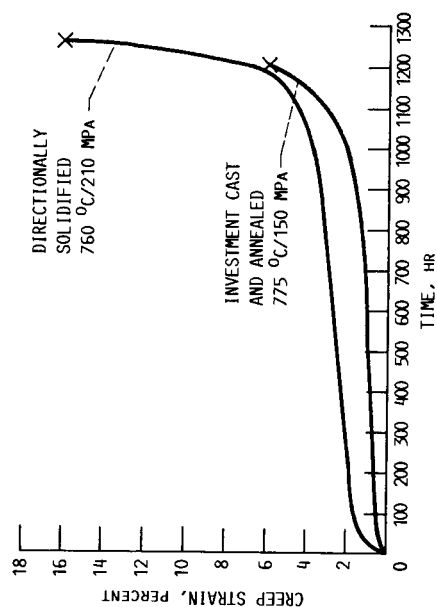


FIGURE 3. - TYPICAL CREEP-RUPTURE CURVES FOR THE DIRECTIONALLY SOLIDIFIED AND THE INVESTMENT CAST NASAUT 4G-A1 TESTED AT THE INDICATED CONDITIONS.

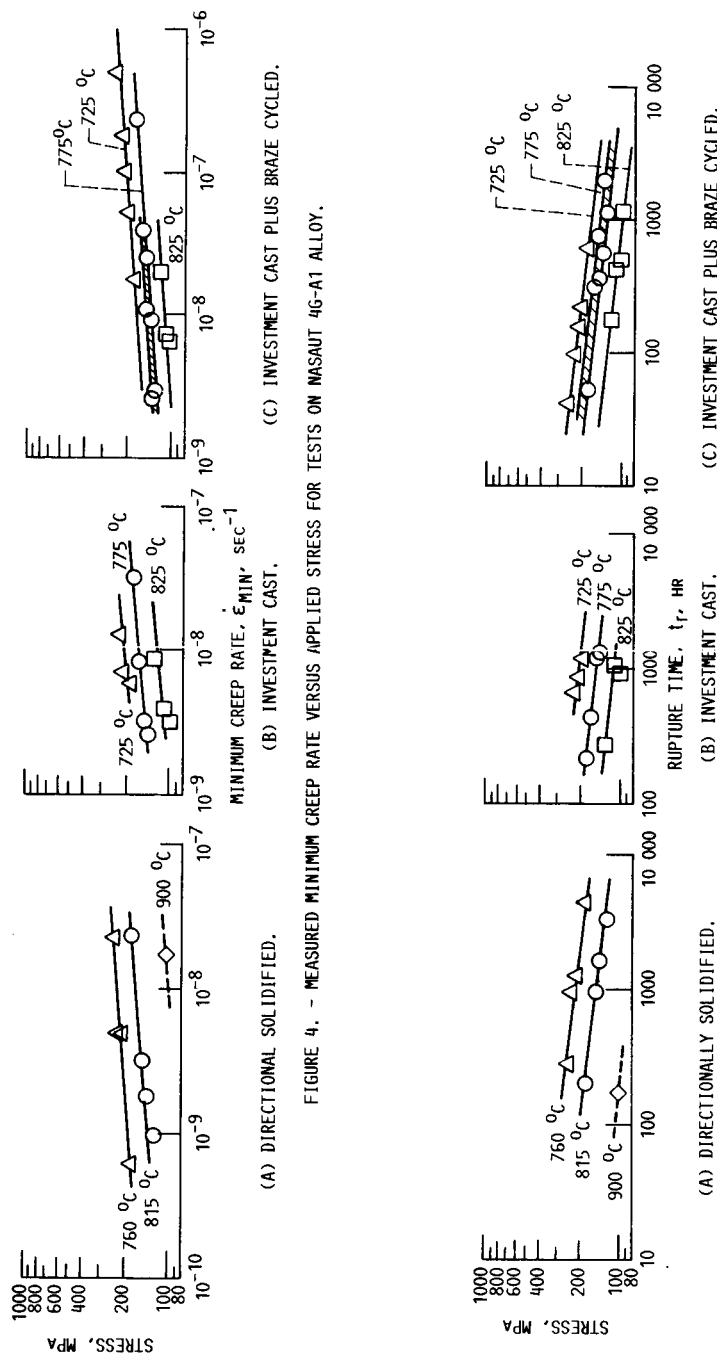


FIGURE 4. - MEASURED MINIMUM CREEP RATE VERSUS APPLIED STRESS FOR TESTS ON NASAUT 4G-A1 ALLOY.

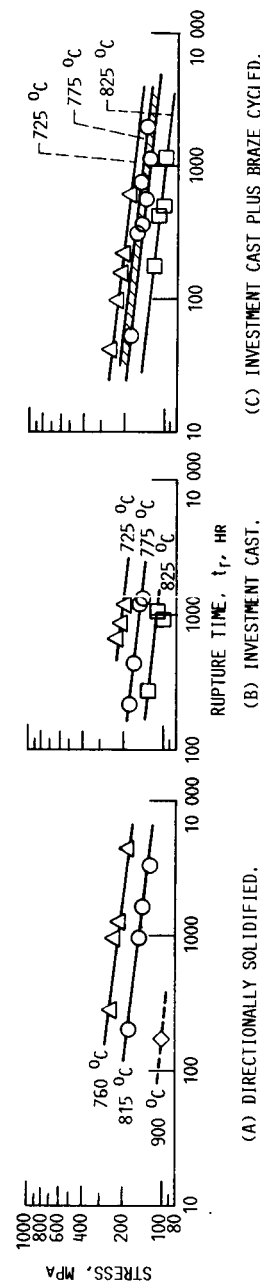
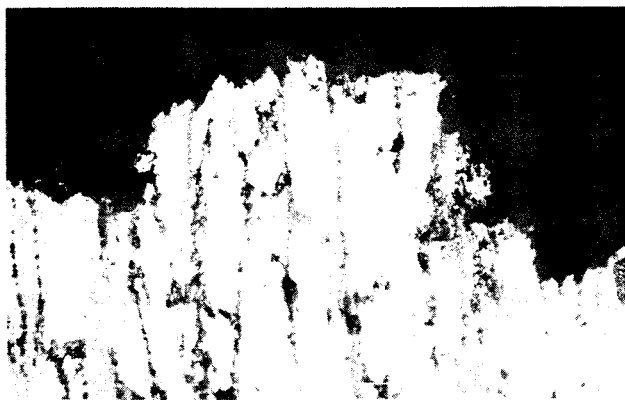
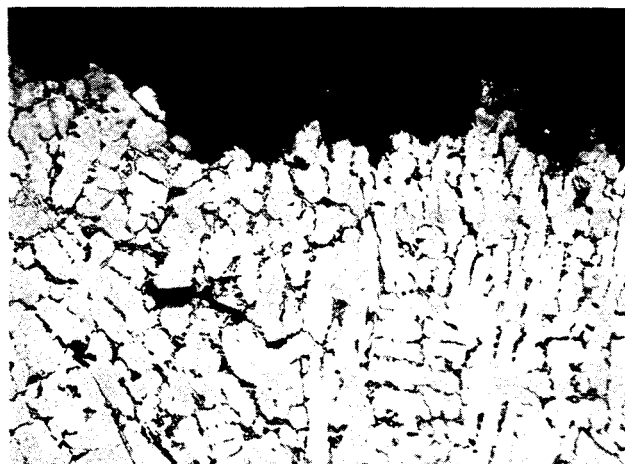


FIGURE 5. - MEASURED RUPTURE TIMES VERSUS APPLIED STRESS FOR TESTS ON NASAUT 4G-A1 ALLOY.



(A) DIRECTIONALLY SOLIDIFIED AND ANNEALED 100 HR AT 650 °C PRIOR TO TESTING AT 815 °C AND 150 MPa (947 HR LIFE).



(B) INVESTMENT CAST AND ANNEALED 20 HR AT 760 °C PRIOR TO TESTING AT 775 °C AND 150 MPa (1210 HR LIFE).



(C) INVESTMENT CAST AND ANNEALED FOR 20 HR AT 760 °C, THEN GIVEN THE BRAZE CYCLE PRIOR TO TESTING AT 775 °C AND 150 MPa (752 HR LIFE).

FIGURE 6. - DUCTILE FRACTURES OF TYPICAL NASAUT 4G-A1 SPECIMENS.

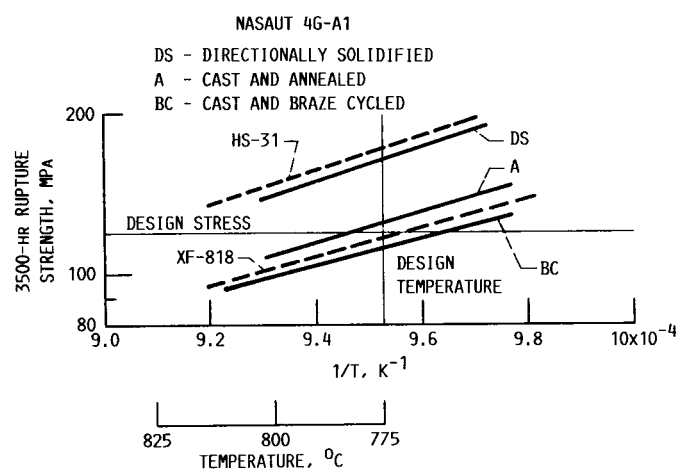


FIGURE 7. - THE TEMPERATURE DEPENDENCY OF 3500-HR RUPTURE STRENGTH FOR CANDIDATE STIRLING ENGINE ALLOYS COMPARED TO THE MOD 1A STIRLING AUTOMOTIVE ENGINE DESIGN CRITERIA.



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